# Visible Light-Promoted Selenylative Spirocyclization of Biaryl Ynones toward the Formation of Selenated Spiro[5.5]trienones

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#### 1. General information

Unless otherwise stated, all commercial materials and solvents were used directly without further purification. <sup>1</sup>H and <sup>13</sup>C NMR spectra were measured on a 400 MHz Bruker spectrometer (<sup>1</sup>H 400MHz, <sup>13</sup>C 100MHz, <sup>19</sup>F NMR 376 MHz), using CDCl<sub>3</sub> (spectra were referenced to the solvent peaks <sup>1</sup>H: residual CDCl<sub>3</sub> = 7.26 ppm, <sup>13</sup>C: CDCl<sub>3</sub> = 77.0 ppm)) as the solvent. High-resolution mass spectra (HRMS) were measured on ESI-TOF. Column chromatography was performed on silica gel (70-230 mesh ASTM) using the reported eluent. Thin-layer chromatography (TLC) was carried out on 4×5 cm plates with a layer thickness of 0.2 mm (silica gel 60 F254). Photochemical reactions were performed with a LED reactor WP-TEC-1020HSL (WATTCAS, China). Starting materials diselenides **2**, biaryl ynones **1** were prepared according to the literatures.<sup>S1,S2</sup>

#### 2. General catalytic procedure for the synthesis of 3



Biaryl ynones 1 (0.2 mmol), diselenides 2 (0.4 mmol), and 1.5 mL MeCN were added in a quartz tube. Then the mixture was stirred at room temperature (rt) for 12 h in the photochemical reactor with 8 W blue LED as light source under  $O_2$  atmosphere. After completion of the biaryl ynones, the solvent was removed under reduced pressure by rotary evaporator. Then, the residue was purified by silica gel column chromatography to give the desired products **3**.



Figure. S1 The WP-TEC-1020HSL photochemical reaction system with the blue light LED

#### 3. Mechanism experiments and large-scale synthesis of 3aa

(1) Trapping experiment with 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO)



Biaryl ynone **1a** (0.2 mmol), diphenyl diselenide **2a** (0.4 mmol), TEMPO (1 mmol, 5.0 equiv) and 1.5 mL MeCN were added in a quartz tube. Then the mixture was stirred at rt for 12 h in the photochemical reactor with 8 W blue LED as light source under  $O_2$  atmosphere. The resulting mixture was monitored by TLC.

#### (2) Trapping experiment with ethene-1,1-diyldibenzene



Biaryl ynone **1a** (0.2 mmol), diphenyl diselenide **2a** (0.4 mmol), ethene-1,1-diyldibenzene (0.2 mmol, 1.0 equiv) and 1.5 mL MeCN were added in a quartz tube. Then the mixture was stirred at rt for 12 h in the photochemical reactor with 8 W blue LED as light source under O<sub>2</sub> atmosphere. The solvent was removed under reduced pressure, purification was performed by flash column chromatography on silica gel with petroleum ether/ethyl acetate as eluent to give the corresponding compound **3aa**, and **4** was detected by HRMS (Figure S2). HRMS (ESI) m/z calcd for C<sub>20</sub>H<sub>17</sub>Se [M+H]<sup>+</sup> 337.0490, found 337.0489.



Figure. S2

(3) Large-scale synthesis of 3aa



Biaryl ynone **1a** (2 mmol), diphenyl diselenide **2a** (4 mmol), and 4 mL MeCN were added in a quartz tube. Then the mixture was stirred at rt for 24 h in the photochemical reactor with 8 W blue LED as light source under  $O_2$  atmosphere. After completion of the biaryl ynones, the solvent was removed under reduced pressure by rotary evaporator. Then, the residue was purified by silica gel column chromatography to give the desired products **3aa**.

#### 4. X-ray data of compound 3ga and 3af

Single crystal suitable for X-ray diffraction experiment was obtained by slow evaporation of DCM/n-hexane (1:10, V/V) solution containing the compound **3ac** and **3af**.



Figure S1. X-ray molecular structure of 3ac with the probability at 50% level.

Table 1 Crystal data and structure refin	nement for 3ac.
Empirical formula	$C_{29}H_{22}O_2Se$
Formula weight	481.42
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	9.6751(8)
b/Å	10.1384(10)
c/Å	13.4857(12)
α /°	107.731(8)
β /°	109.770(8)
γ /°	93.109(7)
Volume/Å3	1167.3(2)
Z	2
ρ calcg/cm3	1.370
μ /mm-1	2.352
F(000)	492.0
Crystal size/mm3	0.16  imes 0.12  imes 0.1
Radiation	CuK $\alpha$ ( $\lambda = 1.54184$ )
$2\Theta$ range for data collection/°	7.418 to 134.15
Index ranges	$\textbf{-11} \leqslant h \leqslant \textbf{7}, \textbf{-12} \leqslant k \leqslant \textbf{12}, \textbf{-14} \leqslant \textbf{l} \leqslant \textbf{16}$
Reflections collected	8277
Independent reflections	4167 [Rint = 0.0409, Rsigma = 0.0542]
Data/restraints/parameters	4167/4/298
Goodness-of-fit on F2	1.064
Final R indexes [I>=2 $\sigma$ (I)]	R1 = 0.0455, wR2 = 0.1041
Final R indexes [all data]	R1 = 0.0626, $wR2 = 0.1160$
Largest diff. peak/hole / e Å-3	0.26/-0.40



Figure S2. X-ray molecular structure of 3af with the probability at 50% level.

#### 5. Characterization of compounds 3

2'-Phenyl-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3aa)<sup>\$3,\$4</sup>



Yellow solid. 76.5 mg, Yield: 82%. mp 205-207 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.27 (d, J = 7.6 Hz, 1H), 7.56 (dt, J = 23.7, 7.3 Hz, 2H), 7.33 – 7.10 (m, 9H), 6.94 (d, J = 7.1 Hz, 2H), 6.76 (d, J = 9.1 Hz, 2H), 6.32 (d, J = 9.1 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.7, 180.6, 158.6, 148.5, 138.0, 137.9, 137.2, 133.4, 133.0, 130.7, 130.1, 130.0, 129.2, 129.0, 128.5, 128.1, 127.8, 127.4, 127.2, 52.6. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>18</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 477.0364, found 477.0366. **2'-Phenyl-3'-(***p***-tolylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione** (3ab)



Yellow solid. 71.1 mg, Yield: 76%. mp 191-193 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.27 (d, J = 7.7 Hz, 1H), 7.56 (dt, J = 22.0, 7.4 Hz, 2H), 7.31 – 7.16 (m, 6H), 6.95 (t, J = 7.1 Hz, 4H), 6.75 (d, J = 9.3 Hz, 2H), 6.32 (d, J = 9.3 Hz, 2H), 2.30 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.7, 180.7, 158.0, 148.5, 138.0, 137.9, 137.4, 137.3, 133.4, 133.3, 130.1, 129.8, 129.1, 128.5, 128.5, 128.0, 127.7, 127.5, 126.8, 52.5, 21.1. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 491.0521, found 491.0523.

3'-((4-Ethylphenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ac)



Yellow solid. 78 mg, Yield: 81%. mp 188-189 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.27 (d, J = 7.7 Hz, 1H), 7.55 (dt, J = 14.9, 7.2 Hz, 2H), 7.31 – 7.24 (m, 2H), 7.22 (d, J = 7.3 Hz, 4H), 6.96 (dd, J = 13.8, 7.5 Hz, 4H), 6.75 (d, J = 9.4 Hz, 2H), 6.31 (d, J = 9.3 Hz, 2H), 2.58 (q, J = 7.5 Hz, 2H), 1.21 (t, J = 7.5 Hz, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.7, 180.7, 157.9, 148.6, 143.6, 138.0, 138.0,

137.5, 133.5, 133.3, 130.1, 130.1, 129.1, 128.6, 128.5, 128.4, 128.1, 127.7, 127.5, 127.0, 52.6, 28.5, 15.5. HRMS (ESI) *m/z* calcd for C<sub>29</sub>H<sub>22</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 505.0677, found 505.0681. **3'-((4-(Tert-butyl)phenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5diene-4,4'-dione (3ad)** 



Yellow solid. 61.2 mg, Yield: 60%. mp 171-173 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.30 (d, J = 7.6 Hz, 1H), 7.57 (dt, J = 19.4, 7.3 Hz, 2H), 7.31 – 7.11 (m, 9H), 6.91 (d, J = 7.6 Hz, 2H), 6.74 (d, J = 9.3 Hz, 2H), 6.31 (d, J = 9.3 Hz, 2H), 1.29 (s, 9H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.8, 157.8, 150.4, 148.6, 137.9, 137.5, 133.3, 133.2, 130.1, 130.0, 129.1, 128.5, 128.4, 128.1, 127.7, 127.5, 126.7, 126.1, 52.6, 34.5, 31.3. HRMS (ESI) *m/z* calcd for C<sub>31</sub>H<sub>26</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 533.0990, found 533.0992.

3'-((4-Methoxyphenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ae)<sup>84</sup>



Yellow solid. 61 mg, Yield: 63%. mp 163-165 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.26 (d, J = 7.5 Hz, 1H), 7.54 (dt, J = 21.9, 7.1 Hz, 2H), 7.27 – 7.21 (m, 6H), 6.94 (d, J = 7.1 Hz, 2H), 6.73 (d, J = 9.3 Hz, 2H), 6.68 (d, J = 7.7 Hz, 2H), 6.29 (d, J = 9.3 Hz, 2H), 3.76 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.9, 159.3, 157.3, 148.6, 137.9, 137.9, 137.8, 135.9, 133.3, 130.0, 129.1, 128.5, 128.0, 127.7, 127.6, 120.3, 114.6, 55.3, 52.5. <sup>77</sup>Se NMR (114 MHz, CDCl<sub>3</sub>)  $\delta$  349.44. HRMS (ESI) m/z calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>3</sub>Se [M+Na]<sup>+</sup> 507.0470, found 507.0472.

# 3'-((4-Fluorophenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3af)<sup>S4</sup>



Yellow solid. 68.9 mg, Yield: 73%. mp 169-170 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.27 (d, J = 7.5 Hz, 1H), 7.57 (dt, J = 14.3, 6.8 Hz, 2H), 7.29 – 7.24 (m, 6H), 6.94 (d, J = 6.9 Hz, 2H), 6.84 (t, J = 7.9 Hz, 2H), 6.74 (d, J = 9.5 Hz, 2H), 6.32 (d, J = 9.6 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ 

184.7, 180.7, 162.4 (d, J = 247.6 Hz), 157.9, 148.4, 138.0, 137.8, 137.5, 135.9 (d, J = 8.0 Hz), 133.5, 130.2, 129.9, 129.2, 128.6, 128.5, 128.1, 127.8, 127.5, 124.8 (d, J = 3.4 Hz), 116.1 (d, J = 21.6 Hz), 52.6. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -113.89. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>FNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 495.0270, found 4795.0272.

2'-Phenyl-3'-(*m*-tolylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ag)



Yellow solid. 73 mg, Yield: 78%. mp 148-149 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.28 (d, J = 7.7 Hz, 1H), 7.56 (dt, J = 22.6, 7.4 Hz, 2H), 7.32 – 7.24 (m, 2H), 7.20 (t, J = 7.5 Hz, 2H), 7.10 (d, J = 7.4 Hz, 1H), 7.06 – 6.97 (m, 3H), 6.93 (d, J = 7.7 Hz, 2H), 6.76 (d, J = 9.6 Hz, 2H), 6.31 (d, J = 9.7 Hz, 2H), 2.23 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.7, 180.7, 158.1, 148.5, 138.6, 138.0, 137.9, 137.4, 133.8, 133.4, 130.4, 130.1, 130.0, 129.1, 128.8, 128.5, 128.4, 128.1, 128.1, 127.7, 127.5, 52.6, 21.3. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 491.0521, found 491.0523. **3'-((3-Chlorophenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ah)** 



Yellow solid. 68.3 mg, Yield: 70%. mp 160-161 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.28 (d, J = 8.9 Hz, 1H), 7.58 (dt, J = 14.1, 7.3 Hz, 2H), 7.33 – 7.26 (m, 2H), 7.27 – 7.12 (m, 5H), 7.07 (t, J = 8.0 Hz, 1H), 6.93 (d, J = 7.3 Hz, 2H), 6.75 (d, J = 10.0 Hz, 2H), 6.33 (d, J = 10.0 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.6, 180.5, 158.7, 148.3, 138.0, 137.6, 137.0, 134.4, 133.6, 132.8, 132.1, 131.2, 130.2, 129.9, 129.9, 129.2, 128.7, 128.5, 128.1, 127.8, 127.5, 127.4, 52.7. <sup>77</sup>Se NMR (114 MHz, CDCl<sub>3</sub>)  $\delta$  372.05. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>ClNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 510.9975, found 510.9977.

3'-((3-Bromophenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ai)



Yellow solid. 72.3 mg, Yield: 68%. mp 163-164 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.30 (d, J = 7.6 Hz, 1H), 7.59 (dt, J = 21.8, 7.3 Hz, 2H), 7.34 – 7.29 (m, 4H), 7.22 (t, J = 7.8 Hz, 3H), 7.01 (t, J = 7.8 Hz, 1H), 6.92 (d, J = 7.6 Hz, 2H), 6.75 (d, J = 9.8 Hz, 2H), 6.33 (d, J = 9.7 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.6, 180.5, 158.5, 148.2, 138.0, 137.6, 137.2, 135.7, 133.6, 132.4, 131.7, 130.4, 130.2, 129.9, 129.2, 128.7, 128.6, 128.1, 127.8, 127.4, 122.6, 52.7. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>BrNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 554.9469, found 554.9471.

2'-Phenyl-3'-((3-(trifluoromethyl)phenyl)selanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3aj)



Yellow solid. 52.2 mg, Yield: 50%. mp 170-172 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.29 (d, J = 7.7 Hz, 1H), 7.59 (dt, J = 22.9, 7.4 Hz, 2H), 7.50 – 7.39 (m, 3H), 7.31 – 7.17 (m, 5H), 6.90 (d, J = 7.5 Hz, 2H), 6.74 (d, J = 9.1 Hz, 2H), 6.32 (d, J = 9.1 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.6, 180.6, 158.6, 148.2, 138.0, 137.6, 137.1, 136.5, 133.6, 131.6, 131.1(q, J = 32.8 Hz) , 130.3, 129.8 (q, J = 3.9 Hz), 129.8, 129.2, 129.2, 128.8, 128.5, 128.1, 127.8, 127.3, 124.07 (q, J = 3.7 Hz), 123.6 (q, J = 273.3 Hz), 52.7. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -62.73. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>17</sub>F<sub>3</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 545.0238, found 545.0239.

2'-Phenyl-3'-(*o*-tolylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ak)



Yellow solid. 49.6 mg, Yield: 53%. mp 159-161 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.28 (d, J = 7.7 Hz, 1H), 7.57 (dt, J = 23.2, 7.4 Hz, 2H), 7.30 (d, J = 8.1 Hz, 2H), 7.25 (d, J = 7.2 Hz, 1H), 7.18 (dd, J = 13.1, 7.2 Hz, 3H), 7.12 – 7.06 (m, 2H), 6.95 – 6.90 (m, 3H), 6.76 (d, J = 9.5 Hz, 2H), 6.32 (d, J = 9.5 Hz, 2H), 2.26 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.7, 180.6, 158.0, 148.5, 139.6, 138.0, 137.8, 137.2, 133.4, 133.2, 131.9, 130.1, 130.0, 129.9, 129.1, 128.5, 128.5, 128.1, 127.7, 127.4, 127.2, 126.4, 52.7, 22.5. HRMS (ESI) *m*/*z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 491.0521, found 491.0524.

3'-((2-Methoxyphenyl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3al)



Yellow solid. 43.6 mg, Yield: 45%, mp 147-148 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.28 (d, J = 7.4 Hz, 1H), 7.57 (dd, J = 15.5, 7.3 Hz, 2H), 7.35 – 7.12 (m, 5H), 7.06 (d, J = 7.2 Hz, 1H), 6.97 (d, J = 6.9 Hz, 2H), 6.76 (t, J = 11.0 Hz, 4H), 6.33 (d, J = 9.4 Hz, 2H), 3.75 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.4, 158.2, 157.3, 148.7, 137.9, 137.9, 136.0, 133.3, 131.9, 130.1, 129.1, 128.6, 128.5, 128.2, 128.0, 127.7, 127.2, 121.3, 120.3, 110.6, 55.6, 52.61. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>3</sub>Se [M+Na]<sup>+</sup> 507.0470, found 507.0467.

3'-(Naphthalen-1-ylselanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3am)



Yellow solid. 41.3 mg, Yield: 41%. mp 157-158 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.31 (d, *J* = 7.5 Hz, 1H), 8.06 (d, *J* = 7.6 Hz, 1H), 7.77 (d, *J* = 7.4 Hz, 1H), 7.69 (d, *J* = 8.2 Hz, 1H), 7.63 – 7.51 (m, 2H), 7.51 – 7.41 (m, 2H), 7.39 (d, *J* = 7.1 Hz, 1H), 7.25 (d, *J* = 7.5 Hz, 1H), 7.15 – 7.03 (m, 2H), 6.97 (t, *J* = 7.4 Hz, 2H), 6.63 (t, *J* = 8.8 Hz, 4H), 6.22 (d, *J* = 9.2 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 181.1, 156.6, 148.6, 138.0, 137.4, 137.2, 133.8, 133.8, 133.6, 133.4, 129.9, 129.8, 129.4, 129.1, 128.7, 128.6, 128.5, 128.2, 128.1, 127.7, 127.5, 127.1, 126.4, 126.1, 125.7, 52.9. HRMS (ESI) *m/z* calcd for C<sub>31</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 527.0521, found 527.0522.

3'-((2-Methoxypyridin-3-yl)selanyl)-2'-phenyl-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5diene-4,4'-dione (3an)



Yellow solid. 48.5 mg, Yield: 50%. mp 161-162 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.29 (d, J = 7.6 Hz, 1H), 7.97 (d, J = 3.3 Hz, 1H), 7.59 (dd, J = 15.4, 7.5 Hz, 2H), 7.39 – 7.13 (m, 5H), 6.95 (d, J = 7.2 Hz, 2H), 6.75 (d, J = 9.4 Hz, 2H), 6.66 (t, J = 5.7 Hz, 1H), 6.33 (d, J = 9.4 Hz, 2H), 3.87 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.7, 180.3, 161.3, 157.9, 148.4, 145.2, 140.8, 138.0, 137.5, 135.3, 133.5, 130.2, 129.8, 129.2, 128.7, 128.5, 128.1, 127.8, 127.2, 117.5, 115.0, 53.8, 52.7. HRMS (ESI) m/z calcd for C<sub>27</sub>H<sub>19</sub>NNaO<sub>3</sub>Se [M+Na]<sup>+</sup> 508.0422, found 508.0423.

3'-(Phenylselanyl)-2'-(*p*-tolyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ba)<sup>S3</sup>



Yellow solid. 66.5 mg, Yield: 71%. mp 159-160 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.26 (d, J = 7.7 Hz, 1H), 7.56 (dt, J = 24.2, 7.4 Hz, 2H), 7.30 (t, J = 7.8 Hz, 3H), 7.21 – 7.13 (m, 3H), 7.03 (d, J = 7.7 Hz, 2H), 6.85 (d, J = 7.7 Hz, 2H), 6.76 (d, J = 9.5 Hz, 2H), 6.33 (d, J = 9.5 Hz, 2H), 2.34 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.5, 158.8, 148.6, 138.4, 138.0, 137.1, 135.3, 133.3, 132.9, 130.8, 130.1, 129.1, 128.9, 128.5, 128.5, 128.1, 127.3, 127.1, 52.7, 21.3. <sup>77</sup>Se NMR (114 MHz, CDCl<sub>3</sub>)  $\delta$  359.52. HRMS (ESI) *m*/*z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 491.0521, found 491.0523.

2'-(4-Methoxyphenyl)-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ca)<sup>S3</sup>



Yellow solid. 64.9 mg, Yield: 67%. mp 163-164 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.27 (d, *J* = 7.8 Hz, 1H), 7.56 (dt, *J* = 14.2, 7.9 Hz, 2H), 7.30 – 7.28 (m, 3H), 7.16 (dt, *J* = 14.1, 4.7 Hz, 3H), 6.88 (d, *J* = 8.6 Hz, 2H), 6.73 (dd, *J* = 9.2, 5.7 Hz, 4H), 6.33 (d, *J* = 9.9 Hz, 2H), 3.80 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.7, 159.6, 158.2, 148.7, 138.0, 137.6, 133.3, 133.0, 130.9, 130.6, 130.1, 129.1, 128.9, 128.8, 128.5, 128.0, 127.2, 113.2, 55.2, 52.9. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>3</sub>Se [M+Na]<sup>+</sup> 507.0470, found 507.0473.

2'-([1,1'-Biphenyl]-4-yl)-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5diene-4,4'-dione (3da)



Yellow solid. 54 mg, Yield: 51%. mp 184-185 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.31 (d, J = 7.7 Hz, 1H), 7.66 – 7.51 (m, 4H), 7.47 (t, J = 7.4 Hz, 2H), 7.40 (d, J = 7.4 Hz, 3H), 7.34 – 7.09 (m, 6H), 6.99 (d, J = 7.8 Hz, 2H), 6.78 (d, J = 9.4 Hz, 2H), 6.35 (d, J = 9.4 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.8, 157.7, 148.6, 141.1, 140.2, 138.0, 137.8, 136.9, 133.4, 133.3, 130.6, 130.2,

130.0, 129.2, 128.9, 128.8, 128.6, 128.1, 127.9, 127.7, 127.3, 127.1, 126.4, 52.7. HRMS (ESI) *m/z* calcd for C<sub>33</sub>H<sub>22</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 553.0677, found 553.0681.

2'-(4-Fluorophenyl)-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ea)



Yellow solid. 65.1 mg, Yield: 69%. mp 178-179 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.29 (d, J = 7.7 Hz, 1H), 7.58 (dt, J = 21.7, 7.4 Hz, 2H), 7.29 – 7.24 (m, 3H), 7.20 – 7.12 (m, 3H), 6.88 (d, J = 6.7 Hz, 4H), 6.72 (d, J = 9.1 Hz, 2H), 6.33 (d, J = 9.1 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.5, 180.7, 162.4 (d, J = 249.1 Hz), 156.8, 148.4, 138.3, 137.9, 133.8 (d, J = 3.6 Hz), 133.5, 133.3, 130.5, 130.2, 129.8, 129.5(d, J = 8.3 Hz), 129.2, 129.0, 128.6, 128.1, 127.4, 114.9 (d, J = 21.8 Hz), 52.6. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)  $\delta$  -112.26. HRMS (ESI) *m*/*z* calcd for C<sub>27</sub>H<sub>17</sub>FNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 495.0270, found 4795.0271.

2'-(4-Chlorophenyl)-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3fa)



Yellow solid. 60.5 mg, Yield: 62%. mp 174-175 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.30 (d, J = 7.7 Hz, 1H), 7.58 (dt, J = 21.0, 7.4 Hz, 2H), 7.33 – 7.17 (m, 4H), 7.15 (d, J = 8.0 Hz, 4H), 6.83 (d, J = 7.9 Hz, 2H), 6.71 (d, J = 9.4 Hz, 2H), 6.33 (d, J = 9.4 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.5, 180.7, 156.3, 148.2, 138.2, 137.8, 136.1, 134.5, 133.5, 133.4, 130.3, 130.3, 129.8, 129.2, 129.0, 128.9, 128.6, 128.1, 128.0, 127.4, 52.5. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>ClNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 510.9975, found 510.9976.

2'-(4-Bromophenyl)-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ga)



Yellow solid. 68.1 mg, Yield: 64%. mp 169-171 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.30 (d, J = 7.6 Hz, 1H), 7.58 (dt, J = 20.6, 7.2 Hz, 2H), 7.31 – 7.12 (m, 8H), 6.77 (d, J = 7.8 Hz, 2H), 6.71 (d, J =

9.3 Hz, 2H), 6.33 (d, J = 9.4 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.47 (s), 180.7, 156.2, 148.2, 138.2, 137.8, 136.6, 133.5, 133.5, 131.0, 130.3, 129.8, 129.2, 129.2, 129.0, 128.6, 128.1, 127.5, 122.8, 52.4. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>BrNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 554.9469, found 554.9472.

4-(4,4'-Dioxo-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-dien-2'yl)benzonitrile (3ha)<sup>S3</sup>



Yellow solid. 54.6 mg, Yield: 57%. mp 180-182 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.31 (d, *J* = 7.6 Hz, 1H), 7.60 (dt, *J* = 21.0, 7.4 Hz, 2H), 7.44 (d, *J* = 7.6 Hz, 2H), 7.28 (d, *J* = 5.9 Hz, 1H), 7.24 – 7.11 (m, 5H), 6.99 (d, *J* = 7.6 Hz, 2H), 6.71 (d, *J* = 9.1 Hz, 2H), 6.33 (d, *J* = 9.2 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.1, 180.5, 154.9, 147.8, 141.8, 138.6, 137.7, 133.8, 133.6, 131.5, 130.5, 130.1, 129.5, 129.4, 129.2, 128.6, 128.6, 128.1, 127.7, 118.1, 112.2, 52.2. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>17</sub>NNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 502.0317, found 502.0320.

3'-(Phenylselanyl)-2'-(*m*-tolyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ia)



Yellow solid. 73 mg, Yield: 78%. mp 167-169 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.27 (d, J = 7.6 Hz, 1H), 7.56 (dt, J = 23.1, 7.2 Hz, 2H), 7.29 (d, J = 6.3 Hz, 3H), 7.18 – 7.07 (m, 5H), 6.76 (t, J = 8.5 Hz, 3H), 6.67 (s, 1H), 6.32 (t, J = 10.3 Hz, 2H), 2.25 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.8, 180.7, 158.7, 148.6, 138.0, 137.9, 137.3, 137.1, 133.4, 133.1, 130.7, 130.0, 129.3, 129.1, 128.9, 128.5, 128.1, 128.0, 127.7, 127.2, 124.5, 52.6, 21.5. HRMS (ESI) *m/z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 491.0521, found 491.0522.

2'-(3-Chlorophenyl)-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ja)



Yellow solid. 60.5 mg, Yield: 62%. mp 171-173 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.30 (d, J = 7.6

Hz, 1H), 7.58 (dt, J = 21.2, 7.0 Hz, 2H), 7.33 – 7.18 (m, 5H), 7.20 – 7.07 (m, 3H), 6.85 (s, 1H), 6.81 (d, J = 7.6 Hz, 1H), 6.72 (t, J = 7.7 Hz, 2H), 6.34 (dd, J = 17.6, 9.7 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.5, 180.7, 155.9, 148.3, 148.0, 139.2, 138.3, 137.8, 133.7, 133.6, 130.4, 130.2, 130.1, 129.8, 129.3, 129.1, 129.1, 128.6, 128.6, 128.1, 127.8, 127.6, 125.8, 52.4. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>ClNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 510.9975, found 510.9974.

7'-Methyl-2'-phenyl-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ka)<sup>83</sup>



Yellow solid. 65.5 mg, Yield: 70%. mp 158-159 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.17 (d, J = 7.6 Hz, 1H), 7.34 (d, J = 7.6 Hz, 1H), 7.31 – 7.10 (m, 8H), 7.05 (s, 1H), 6.93 (d, J = 5.9 Hz, 2H), 6.75 (d, J = 9.3 Hz, 2H), 6.32 (d, J = 9.3 Hz, 2H), 2.40 (s, 3H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.9, 180.4, 158.2, 148.7, 144.5, 138.1, 137.9, 137.3, 132.9, 130.8, 130.3, 130.1, 129.0, 128.6, 128.5, 128.2, 127.7, 127.5, 127.1, 52.6, 21.8. HRMS (ESI) *m*/*z* calcd for C<sub>28</sub>H<sub>20</sub>NaO<sub>2</sub>Se [M+Na]<sup>+</sup> 491.0521, found 491.0520.

7'-Chloro-2'-phenyl-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3la)



Yellow solid. 70.3 mg, Yield: 72%. mp 151-153 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.21 (d, J = 7.4 Hz, 1H), 7.50 (d, J = 8.1 Hz, 1H), 7.34 – 7.10 (m, 9H), 6.93 (d, J = 5.5 Hz, 2H), 6.74 (d, J = 9.0 Hz, 2H), 6.35 (d, J = 9.0 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.3, 179.8, 158.2, 147.6, 140.1, 139.8, 137.7, 137.2, 133.1, 130.6, 130.5, 130.2, 129.8, 129.0, 128.7, 128.4, 128.0, 127.8, 127.4, 127.4, 52.2. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>ClNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 510.9975, found 510.9975. **6'-Fluoro-2'-phenyl-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3ma)**<sup>83</sup>



Yellow solid. 64.2 mg, Yield: 68%. mp 161-162 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.30 (s, 1H),

7.35 – 7.10 (m, 9H), 6.97 – 6.92 (m, 3H), 6.75 (d, J = 9.0 Hz, 2H), 6.34 (d, J = 9.0 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 184.3, 179.5, 165.6 (d, J = 256.9 Hz), 158.3, 147.8, 141.1 (d, J = 8.3 Hz), 137.8, 137.2, 133.1, 131.7 (d, J = 9.5 Hz), 130.5, 130.4, 129.0, 128.6, 127.8, 127.4, 127.3, 126.6 (d, J = 2.4 Hz), 117.2 (d, J = 22.1 Hz), 114.7 (d, J = 23.3 Hz), 52.4. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) δ -103.05. HRMS (ESI) m/z calcd for C<sub>27</sub>H<sub>17</sub>FNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 495.0270, found 4795.0272.

6'-Chloro-2'-phenyl-3'-(phenylselanyl)-4'H-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3na)



Yellow solid. 63.4 mg, Yield: 65%. mp 157-158 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.23 (s, 1H), 7.54 (d, *J* = 8.3 Hz, 1H), 7.33 – 7.12 (m, 9H), 6.94 (d, *J* = 6.9 Hz, 2H), 6.72 (d, *J* = 9.7 Hz, 2H), 6.33 (d, *J* = 9.7 Hz, 2H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  184.4, 179.5, 158.7, 147.8, 137.7, 137.1, 136.3, 135.7, 133.5, 133.2, 131.3, 130.4, 129.8, 129.1, 128.7, 128.2, 127.9, 127.4, 127.4, 52.2. HRMS (ESI) *m/z* calcd for C<sub>27</sub>H<sub>17</sub>ClNaO<sub>2</sub>Se [M+Na]<sup>+</sup> 510.9975, found 510.9978.

#### 6. References

- [S1] (a) Green Chem., 2019, 21, 3547. (b) Adv. Synth. Catal., 2020, 362, 3485.
- [S2] (a) Org. Lett., 2018, 20, 2988. (b) Org. Lett., 2021, 23, 2548.
- [S3] J. Org. Chem., 2021, 86, 17071.
- [S4] J. Org. Chem., 2022, 87, 4273.

### 7. Copies of the <sup>1</sup>H, <sup>13</sup>C, <sup>19</sup>F and <sup>77</sup>Se NMR Spectra

<sup>1</sup>H NMR spectrum of **3aa** 





#### <sup>1</sup>H NMR spectrum of **3ab**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)

#### <sup>1</sup>H NMR spectrum of **3ac**



17

80 70

90

60 50

30 20 10

0 -10

40

180 170 160 150 140 130 120 110 100 fl (ppm)

210 200 190

#### <sup>1</sup>H NMR spectrum of **3ad**







<sup>1</sup>H NMR spectrum of **3ae** 

CQ-3



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)

Ö







#### <sup>77</sup>Se NMR spectrum of **3ae**

3ae-77se.2.fid

--349.44







<sup>19</sup>F NMR spectrum of **3af** 

cq-6

O Se Ph

<sup>19</sup>F NMR (376MHz, CDCl<sub>3</sub>)

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 fl (ppm)

#### <sup>1</sup>H NMR spectrum of **3ag**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)





#### <sup>1</sup>H NMR spectrum of **3ah**

cq-11

# (2) (2) (3) (4) (5) (4) (5) (5) (4) (5) (5) (6) (7)





<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)







#### <sup>77</sup>Se NMR spectrum of **3ah**

3ah-77se.1.fid

-372.05









26

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 fl (ppm)

#### <sup>1</sup>H NMR spectrum of **3ak**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)







#### <sup>1</sup>H NMR spectrum of **3al**





#### <sup>1</sup>H NMR spectrum of **3am**







#### <sup>1</sup>H NMR spectrum of **3an**



#### <sup>1</sup>H NMR spectrum of **3ba**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)

ö









#### <sup>77</sup>Se NMR spectrum of **3ba**

3ba-77se.1.fid



















#### <sup>1</sup>H NMR spectrum of **3ea**

170 160 150 140

CQ-19

# 

110 100 fl (ppm) 60 50





<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)



#### <sup>13</sup>C NMR spectrum of **3ea**



10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 fl (ppm)

#### <sup>1</sup>H NMR spectrum of **3fa**



## <sup>13</sup>C NMR spectrum of **3fa**



#### <sup>1</sup>H NMR spectrum of **3ga**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)







#### <sup>1</sup>H NMR spectrum of **3ha**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)







#### <sup>1</sup>H NMR spectrum of **3ia**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)

ö





#### <sup>1</sup>H NMR spectrum of **3ja**



<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)









41

80 70

60 50

90

40 30 20 10

0 -10

210 200 190 180 170 160 150 140 130 120 110 100 f1 (ppm)

#### <sup>1</sup>H NMR spectrum of **3la**







<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)





#### <sup>1</sup>H NMR spectrum of **3ma**







<sup>1</sup>H NMR (400MHz, CDCl<sub>3</sub>)



## <sup>13</sup>C NMR spectrum of **3ma**

cq=28=5	0 0	400	0070000000000	
	04	50 m cv	10400000000000	2
	40	0400	N00000000000	4
	~1 00	0 Ú Ú	40000000	2
			~~~~~~	LO LO
		111		



#### <sup>19</sup>F NMR spectrum of **3ma**

cq-28-5





<sup>19</sup>F NMR (376MHz, CDCl<sub>3</sub>)



<sup>1</sup>H NMR spectrum of **3na** 



